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DEPARTMENT OF THE ARMY
Fort Detrick
Frederick, Maryland

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A method for rapid and serial analysis of the two alkaloid groups in ergot.

by M. Poehm.

Mitteilungen des Chemischen Forschungsinstitutes der Wirtschaft Oesterreichs, 8: 33-36 (1954). (Partial translation).

Operating procedure.

1. Total alkaloid content: 2.0 g of finely pulverized ergot (sieve with a mesh-interval of 0.3 mm) are mixed in a 150 ml medical bottle with 2.5 ml of 5% ammonia and 100.0 ml analytically pure ether (pipette). The tightly closed bottle is then shaken for two hours by machine. Aliquot parts are now removed by pipette from the clear ether solution above the drug; this is accomplished by compressing the solution into a transfer pipette with capillaries, utilizing a glass T piece through which the pipette is inserted. The T piece is attached to the bottle with the aid of a perforated cork; a rubber washer seats the pipette. It is advantageous to use standard bottles and pipettes.

5 ml of removed ether solution are placed in a ground test tube of 30 ml capacity, two small glass beads are added and the tube is placed in an electrically heated pot with water at 45°C. The ether disappears within a few minutes. The residue in the test tube is now supplied, in sequence, with 4 ml of 2% aqueous tartaric acid solution, 8 ml p-dimethylaminobenzaldehyde reagent (Brit. Ph.) and about 10 cm^2 filter paper (Whatman No. 1, Schleicher and Schuell 2043 a). The closed tube is now shaken by machine for 15 minutes, 10 ml of ligroin (boiling point 35-40°C) are added and shaken for 10 minutes more. The ligroin itself should not produce coloration with the sulfuric acid reagent (blank test). The contents of the test tube are then placed on a sand filter, which is constructed in the following manner. A wad of glass wool is inserted at the juncture of a funnel (not too tightly) and covered with the purest, fine quartz sand. The filtrate consists of about 10 ml of a usually turbid, blue solution; the filter paper, separated into fibers by shaking, is deposited as a relatively solid adhering layer above the sand; the supernatant ligroin is poured off and discarded. The first filtrate is fed through once more, yielding an optically clear filtrate already after the passage of 1-2 ml. Its extinction is determined photometrically. Measurement is accomplished best in a spectral photometer (Beckmann quartz spectrophotometer or a similar instrument) with light of 630 mμ wave length (see 1). A mixture of two parts of Secale reagent and one part water serves as a comparative solution.

Computation: Total alkaloid content (mg % computed as ergotamine base) =

$$= \frac{E/d \times 10.78 \times 4 \times 20}{2} = 431.2 \times E/d$$

d = thickness of the cell in cm (usually 1 cm)
E = directly measured extinction, or extinction computed from the measured percentual transparency.

II. Analysis of water-soluble alkaloids:

10 ml of the ether extract are extracted as in I. and evaporated in a ground test tube. The residue is mixed with 5 ml benzene and, after shaking briefly, with 5 ml of 2% aqueous ammonsulfate solution. The closed tube is now gently moved from side to side in a horizontal position and the benzene is renewed three times after 2 minutes of movement each by drawing off the supernatant benzene solution with a capillary (water stream pump). The benzene solution is then discarded. Next, the solution is shaken in the same manner with ca. 10 ml of ligroin. A defined quantity is removed from the ammonsulfate, which by now only contains water-soluble alkaloids, and is mixed with precisely the two-fold volume of Secale reagent. Photometric measurement follows as above.

Computation: Content of ergometrine / ergometrinine (mg %) =

$$= \frac{E/d \times 6.03 \times 5 \times 10}{2} = 150.75 \times E/d.$$

III. Water-insoluble alkaloids: After arithmetical transposition of the content of water-coluble alkaloids to the same computational basis as that of the total alkaloid content (multiplication by 1.786 = ratio of the mole weight ergotamine:ergometrine), the content of water-insoluble alkaloids (computed in mg % as ergotamine base) is determined as the difference of the two data.

Area of utilization:

We use the method for the qualitative analysis of ergot, ergot drugs from which fat has been removed, various degrees of extracted ergot powder, but also of prescriptions of ergot such as dry and Spissa extracts and of preparations of ergot alkaloids (tablets, lozenges, etc.)

NOTE

(1) M. Poehm, Mitt. chem. Forsch.-Inst. Wirtsch. Oesterr. 7, 121 (1953).